## **Test Procedure for**

# ROLLING THIN FILM OVEN TEST FOR ASPHALT BINDERS



**TxDOT Designation: Tex-541-C** 

Effective Date: June 2000

### 1. SCOPE

- 1.1 This procedure describes the Rolling Thin Film Oven (RTFO) Test, used to simulate the short-term aging of asphalt binders that occurs during the hot-mixing process. This procedure is similar but not identical to AASHTO T 240. Use this test to calculate the change in sample mass on heating, but its main function is to produce an aged material for analysis by other suitable means. Included in this test procedure is a method of calibrating the air flow meters for the oven.
- 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

#### 2. APPARATUS

- 2.1 *Rolling thin film oven (RTFO)*, with a flow meter and thermometer as specified in AASHTO T 240, prepared in accordance with Section 3.
- 2.2 Eight sample containers (RTFO bottles), as specified in AASHTO T 240.
- 2.3 *Balance*, conforming to AASHTO M 231, Class B if measuring mass change, Class G2 if not measuring mass change.
- 2.4 *Spatula or other tool*, sufficiently shaped to scrape material from the inside of the RTFO bottles.
- 2.5 Sample dishes, tins, or cups, for collecting aged material at the end of the test.

#### 3. PREPARATION

- 3.1 Make the following preparations to the RTFO well in advance of any testing. Repeat this procedure as part of periodic calibration or after any extended period of disuse.
- Ensure that the sample bottles in the oven's carriage are level. Ideally, the bottles should be level if the oven is level, but this may not be true.

- 3.3 Position the air nozzle outlet so that it is 6.4 mm (0.25 in.) from the mouth of a sample container in the oven carriage and so that, as the carriage turns, the nozzle passes across the center of the container's mouth. 3.4 Position the thermometer so that its bulb is with 25 mm (1 in.) of the same height as the center of the carriage. 3.5 Preheat the oven for at least 2 hr. with the temperature control set at 163°C (325°F), with the air on, and with the flow rate set at 4 L/min. 3.6 Load the oven (empty bottles are acceptable) and start the carriage. 3.7 Wait for the temperature to stabilize. 3.8 If the temperature does not finally return to  $163 \pm 0.5$  °C ( $325 \pm 1$  °F), adjust the temperature setting and repeat Section 3.7. 4. RTFO TEST PROCEDURE
- 4.1 Determine the number of sample bottles to use. A good estimate is one bottle per 25 g of material needed from the test; add two more bottles if measuring mass change.
- 4.2 Verify that the airflow to the oven is on and set to  $4 \pm 0.2$  L/min.
- 4.3 Verify that the temperature is set to the appropriate temperature as determined in Section 3 and that the oven is equilibrated at that temperature.
- 4.4 Heat the asphalt binder sample in a 163°C (325°F) oven (using the RTFO is acceptable) until it is completely fluid and pourable.

**Note 1**—Use other heating and pouring temperatures if recommended by the material's manufacturer.

- 4.5 Remove the sample from the oven and briefly stir with a clean spatula.
- 4.6 If measuring mass change, weigh the two empty mass-change bottles to the nearest 0.001 g and record the results.
- 4.7 Pour  $35 \pm 0.5$  g of asphalt into a sample bottle.
- 4.8 Place the bottle on its side and roll it over to spread the material around the inside.
- 4.9 Repeat Sections 4.7 and 4.8 for the second bottle.
- 4.10 Set the mass-change bottles aside and allow them to cool.
- 4.11 Pour  $35 \pm 0.5$  g of asphalt into a sample bottle.
- 4.12 Place the bottle on its side and roll it over to spread the material around the inside.

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4.13 Repeat Sections 4.11 and 4.12 for each sample bottle.

- When the mass-change bottles have cooled to a safe handling temperature, weigh to the nearest 0.001 g and record the results.
- 4.15 As soon as possible after pouring, load the sample bottles in the sample carriage. If not using all spaces on the sample carriage, arrange the sample bottles in a symmetric manner around the carriage.
- 4.16 If aging other samples in the same oven, allow the current bottles to sit in the oven with the carriage off until the rest of the samples are loaded. However, starting the test within 5 min. of loading any samples into the carriage and evenly distributing all bottles around the carriage is mandatory.
- 4.17 Begin timing the test when all samples are loaded, the oven door is closed, and the carriage is rotating at  $15 \pm 0.2$  rpm.
- 4.18 Observe the temperature in the beginning of the test. If the temperature does not return to  $163 \pm 0.5$  °C ( $325 \pm 1$  °F) within 10 min. of start, the test is invalidated.
- 4.19 Keep the samples in the oven, on temperature, with airflow on and the carriage rotating for  $85 \pm 5$  min.
- 4.20 If measuring mass change, remove the previously weighed bottles from the oven, and place them on their sides to cool in the same area where they cooled after pourup.
- 4.21 Remove one non mass-change bottle from the oven.
- 4.22 Close the door and leave the carriage off.
- 4.23 Transfer the residue from the bottle into a dish, tin, or cup, first by pouring as much as possible, and then by scraping out as much as is practical.
- 4.24 Transfer material from all remaining bottles into sample containers. Combine residue from bottles that came from the same initial sample into one container.
- 4.25 Cool the residue sample to room temperature.
- When mass-change bottles have cooled to a safe handling temperature, weigh them to the nearest 0.001 g, record the result, and discard the residue.
  - **Note 2**—Try to duplicate the cooling time and handling temperature used in Section 4.14 as closely as possible.
- 4.27 Calculate the mass change according to Section 5 and report to the nearest 0.01%.
- 4.28 Before performing further testing on residue samples, reheat the material in a 163°C (325°F) (or manufacturer's recommended temperature) oven to a pourable temperature and stir thoroughly with a clean spatula.

#### 5. CALCULATIONS

5.1 Calculate the mass change of the sample as a percentage of the original mass:

$$\Delta M = \frac{M_2 - M_1}{M_1 - M_h} \times 100\%$$

Where:

 $\Delta M = \text{mass change}$ 

 $M_1$  = initial mass

 $M_2$  = the final mass of bottle and asphalt

 $M_b$  = empty weight of bottle.

## 6. CALIBRATING FLOW METERS

- Use this procedure to calibrate air flow meters on rolling thin film ovens (RTFOs) assuring that the meter will accurately measure the airflow at 4 L/min., as specified.
- As an alternative to this procedure, use a standard flow meter or commercial calibrator, provided it is National Institute of Standards and Technology (NIST) traceable.
- 6.3 *Apparatus:*
- 6.3.1 *Flexible (Tigon) tubing,* several feet.
- 6.3.2 *Open-top water container*, fairly large, with a depth of at least about 15 cm (6 in.) and a width of at least about 45 cm (18 in.)
- 6.3.3 *Graduated cylinder*, with a capacity of at least 2 L (1/2 gal.), readable with an accuracy of  $\pm$  5%.
- 6.3.4 *Certified Timer.*
- 6.4 *Procedure:*
- 6.4.1 Cool the RTFO to room temperature.
- Attach one end of the flexible tubing to the air nozzle inside the oven.

**Note 3**—Do this with or without using some type of fitting, as long as there is a good seal.

- Fill the large container about 90% full of water.
- 6.4.4 Fill the graduated cylinder with water.

- 6.4.5 Either by submerging the graduated cylinder or by sealing its top, invert the graduated cylinder so that its open end is below the surface of the water in the large container and the graduated cylinder is still filled with water.
- Record the volume of air in the graduated cylinder.
- 6.4.7 Set the flow meter for the desired flow.
- 6.4.8 Perform the following actions in rapid succession:
- Move the open end of the flexible tubing underneath the submerged end of the graduated cylinder so that the air coming from the tubing bubbles up into the cylinder.
- 6.4.8.2 Start the timer when the bubbles begin to flow into the cylinder.
- After at least 1 L (1 qt.) and before 2 L (1/2 gal.) of water has been displaced from the cylinder, remove the tubing and stop the timer.
- 6.4.10 Record:
  - volume of air in the graduated cylinder,
  - elapsed time from the timer,
  - barometric pressure, and
  - room temperature.
- 6.4.11 Calculate the approximate flow rate by dividing the volume by the time.
- 6.4.12 Correct the flow rate to standard temperature and pressure (25°C, 760 mm Hg).

**Note 4**—Section 6.5 lists one method of calculating the temperature and pressure corrections.

- 6.4.13 If the flow rate is not equal to 4 L/min., repeat the procedure at several different flow meter settings, and use the data to generate a calibration curve relating the flow meter setting to the actual flow rate.
- 6.5 *Calculations:*
- 6.5.1 Since the room temperature and pressure should normally be fairly close to standard conditions, and since the measured gas is air, the assumption of ideal gas behavior should be satisfactory for this calculation.
- Knowing this, first correct the gas pressure for the height of the water column. To do this, find the average height in inches of the water column in the graduated cylinder. This is height above the surface of the water in the large container. Multiply this number by 1.87 to convert to mm Hg. Now the pressure of the air is the barometric pressure minus this correction. Neglect this correction, if desired, as it will not introduce a large error. In this case, simply use the barometric pressure in the equations below.

Relate the measured volume to the standard volume by a simple equation of proportion:

$$\frac{P_b V}{T_r} = \frac{P_s V_s}{T_s}$$

Where:

 $P_b$  = corrected air pressure

 $T_r$  = absolute temperature of the room

V = measured volume (ending volume minus beginning volume).

The right-hand side represents the pressure, absolute temperature, and volume at standard conditions. Rearranging this gives:

$$V_s = V \frac{T_s P_b}{T_r P_s} = V \frac{P}{T + 273} \left(\frac{298}{760}\right)$$

Where:

P =corrected air pressure, mm Hg

 $T = \text{room temperature, } ^{\circ}\text{C.}$ 

Volumes are in mL, so the standard volume divided by the measured time in sec. gives the flow rate in mL/sec. Convert this to L/min., resulting in:

$$F = \left(\frac{V}{t}\right) \left(\frac{P}{T + 273}\right) \left(\frac{298}{760}\right) \left(\frac{60}{1000}\right) = 0.0235 \left(\frac{V}{t}\right) \left(\frac{P}{T + 273}\right)$$

Where:

F =flow rate, L/min.

P =corrected air pressure, mm Hg

 $T = \text{room temperature, } ^{\circ}\text{C}$ 

V = measured volume of air, mL

t = measured time, sec.